

Fig.1

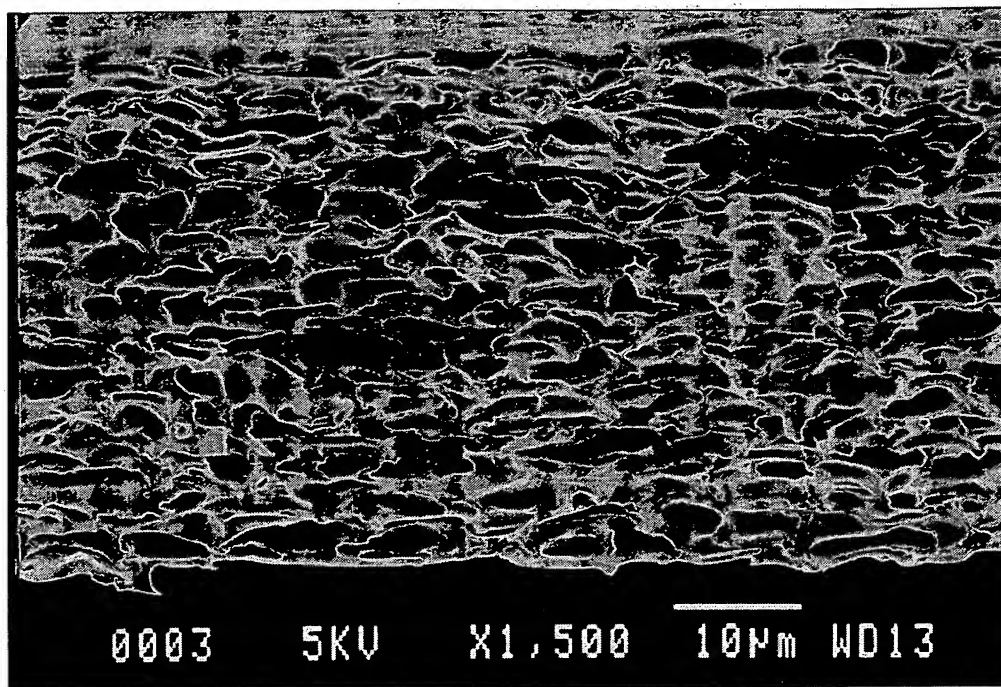


Fig.2

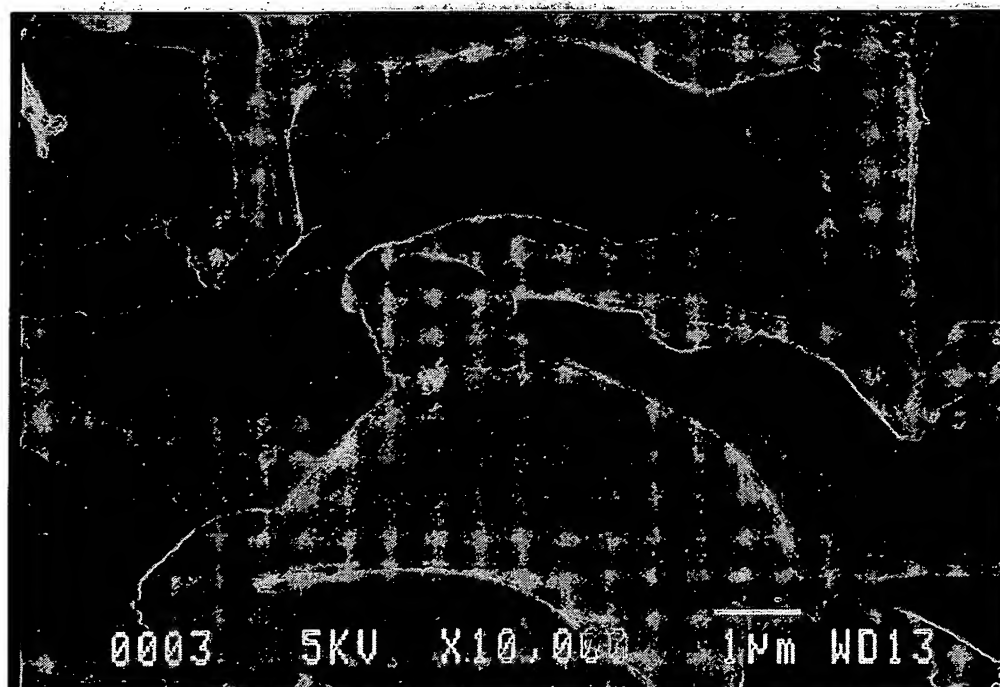


Fig.3

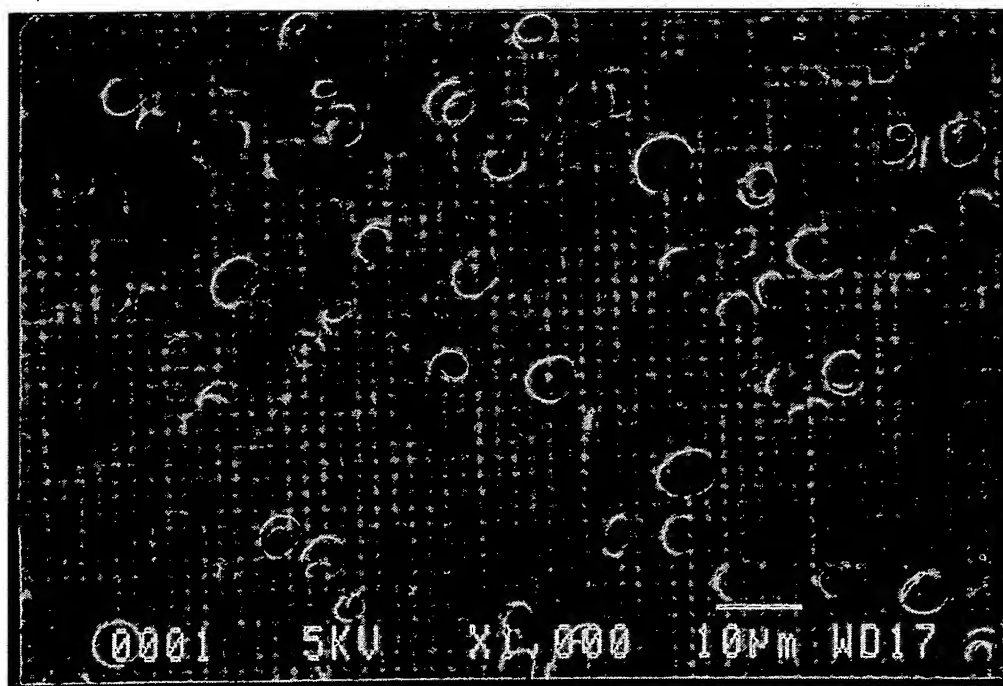
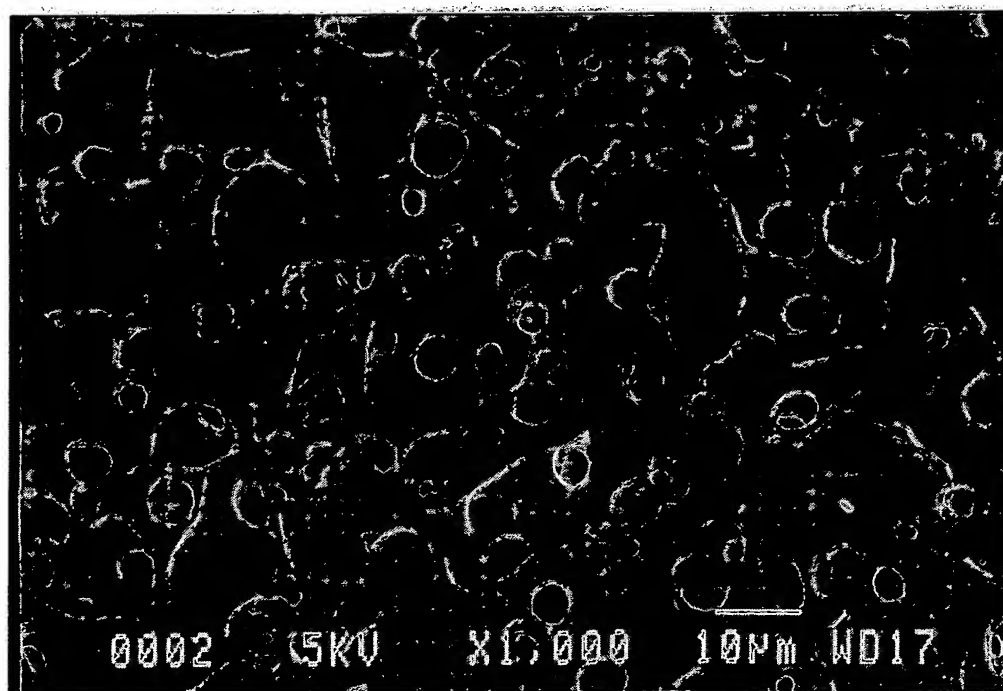


Fig.4





IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of
Shigeru YAO, et al.

Serial No.: 10/785,413 Group Art Unit: 1771

Filed: February 25, 2004 Examiner: VO, Hai

For: POROUS INSULATING FILM AND ITS LAMINATES

DECLARATION UNDER 37 C.F.R. 1.132

Assistant Commissioner for Patents
Alexandria, VA 22313

Sir:

I, Shyusei OHYA, c/o Polymer Research Laboratory,
Corporate Research & Development, Ube Industries, Ltd., 8-
1, Goiminamikaigan, Ichihara-shi, Chiba, Japan do hereby
declare:

That I am a co-inventor of the invention in the
above-identified U.S. application (hereinafter referred to
as "present invention" for brevity) and hence I am fully
familiar therewith;

That I have read and am fully familiar with the
art cited against the claims of the above-identified U.S.
application (hereinafter referred to as "present
application for brevity);

That, in conjunction with the other co-inventors,

I carried out the working examples including comparative examples set forth in the specification of the present application, and the results were as set forth therein;

That I carried out the following additional working.

Additional Working

Reference Example A

Using 3,3',4,4'-biphenyltetracarboxylic dianhydride (s-BPDA) as the tetracarboxylic acid component, 4,4'-diaminodiphenylether (DADE) as the diamine component, these were dissolved in N-methyl-2-pyrrolidone (NMP) with the DADE at a molar ratio of 1.00 with respect to the s-BPDA and the monomer components at a total weight of 9.0 wt%, and polymerized at a temperature of 40°C for 15 hours to obtain a polyimide precursor solution. The solution viscosity of the polyimide precursor solution was 1000 poises.

Reference Example B

Using 3,3',4,4'-biphenyltetracarboxylic dianhydride (s-BPDA) as the tetracarboxylic acid component, 4,4'-diaminodiphenylether (DADE) as the diamine component, these were dissolved in N-methyl-2-pyrrolidone (NMP) with the DADE at a molar ratio of 1.00 with respect to the s-BPDA and the monomer components at a total weight of 6.0 wt%, and polymerized at a temperature of 40°C for 15 hours to obtain a polyimide precursor solution. The solution viscosity of the polyimide precursor solution was 640 poises.

Additional Example A

The polyimide precursor solution obtained in Reference Example A was cast onto a mirror polished stainless steel substrate to a thickness of about 200 μm , and then the surface thereof was covered with a polyolefin fine porous film (UP-3025, product of Ube Industries, Ltd.) with a resistance to passage of air of 550 sec/100 cc as a solvent substitution rate adjustor, without producing wrinkles. The laminate was immersed in 1-pentanol for 100 minutes and solvent substitution with the solvent substitution rate adjustor for precipitation of and pore formation in the polyimide precursor.

The precipitated polyimide precursor porous film was immersed in water for 15 minutes and then released from the stainless steel substrate and solvent substitution rate adjustor and affixed to a pin tenter for heat treatment in nitrogen atmosphere at 300°C for 10 minutes, to obtain a porous polyimide film having a imidization ratio of not lower than 80%.

Scanning electron microscope photographs of the surface and a cross-section of the obtained porous polyimide film are shown in the attached Figs. A and B. It is seen from Fig. B of the cross-sectional photograph that the film contains continuous channels that reach to both surfaces of the film in a linear fashion.

The properties of the obtained film were measured in the same manner as described in the specification of the

present application.

The average pore size at surface was determined from the scanning electron microscope photograph of the surface of the porous polyimide film, i.e. Fig. A, by measuring the pore areas of the open portions of 50 or more and calculating the average pore size from the average pore area value supposing that the open portions have a circular shape.

The results were as follows:

Film thickness: 34 μm

Porosity: 33%

Resistance to passage of air: 605 sec/100 cc

Average pore size at surface: 2.4 μm

Heat shrinkage factor: 0.28%

Additional Example B

The polyimide precursor solution obtained in Reference Example A was cast onto a mirror polished stainless steel substrate to a thickness of about 200 μm , and then the surface thereof was covered with a polyolefin fine porous film (UP-3025, product of Ube Industries, Ltd.) with a resistance to passage of air of 550 sec/100 cc as a solvent substitution rate adjustor, without producing wrinkles. The laminate was immersed in 1-haxanol for 120 minutes and solvent substitution with the solvent substitution rate adjustor for precipitation of and pore formation in the polyimide precursor.

The precipitated polyimide precursor porous film was

immersed in water for 30 minutes and then released from the stainless steel substrate and solvent substitution rate adjustor and affixed to a pin tenter for heat treatment in nitrogen atmosphere at 300°C for 10 minutes, to obtain a porous polyimide film having a imidization ratio of not lower than 80%.

Scanning electron microscope photographs of the surface and a cross-section of the obtained porous polyimide film are shown in the attached Figs. C and D. It is seen from Fig. D of the cross-sectional photograph that the film contains continuous channels that reach to both surfaces of the film in a linear fashion.

The properties of the obtained film determined in the same manner as described in Additional Example A were as follows:

Film thickness: 34 μm

Porosity: 34%

Resistance to passage of air: 740 sec/100 cc

Average pore size at surface: 2.8 μm

Heat shrinkage factor: 0.26%

Additional Example C

The polyimide precursor solution obtained in Reference Example B was cast onto a mirror polished stainless steel substrate to a thickness of about 200 μm , and then the surface thereof was covered with a polyolefin fine porous film (UP-3025, product of Ube Industries, Ltd.) with a resistance to passage of air of 550 sec/100 cc as a solvent substitution rate adjustor, without producing wrinkles.

The laminate was immersed in methanol for 10 minutes and solvent substitution with the solvent substitution rate adjustor for precipitation of and pore formation in the polyimide precursor.

The precipitated polyimide precursor porous film was immersed in water for 30 minutes and then released from the stainless steel substrate and solvent substitution rate adjustor and affixed to a pin tenter for heat treatment in nitrogen atmosphere at 300°C for 10 minutes, to obtain a porous polyimide film having a imidization ratio of not lower than 80%.

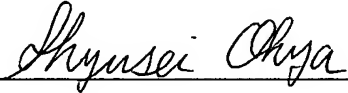
A scanning electron microscope photograph of the surface of the obtained porous polyimide film is shown in the attached Fig. E. It was confirmed by the observation of a cross-section of the obtained polyimide film that the film contains continuous channels that reach to both surfaces of the film in a linear fashion.

The properties of the obtained film determined in the same manner as described in Additional Example A were as follows:

Film thickness: 20 μm
Porosity: 38%
Resistance to passage of air: 36 sec/100 cc
Average pore size at surface: 0.14 μm
Heat shrinkage factor: 0.29%

I, the undersigned declarant, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and; further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

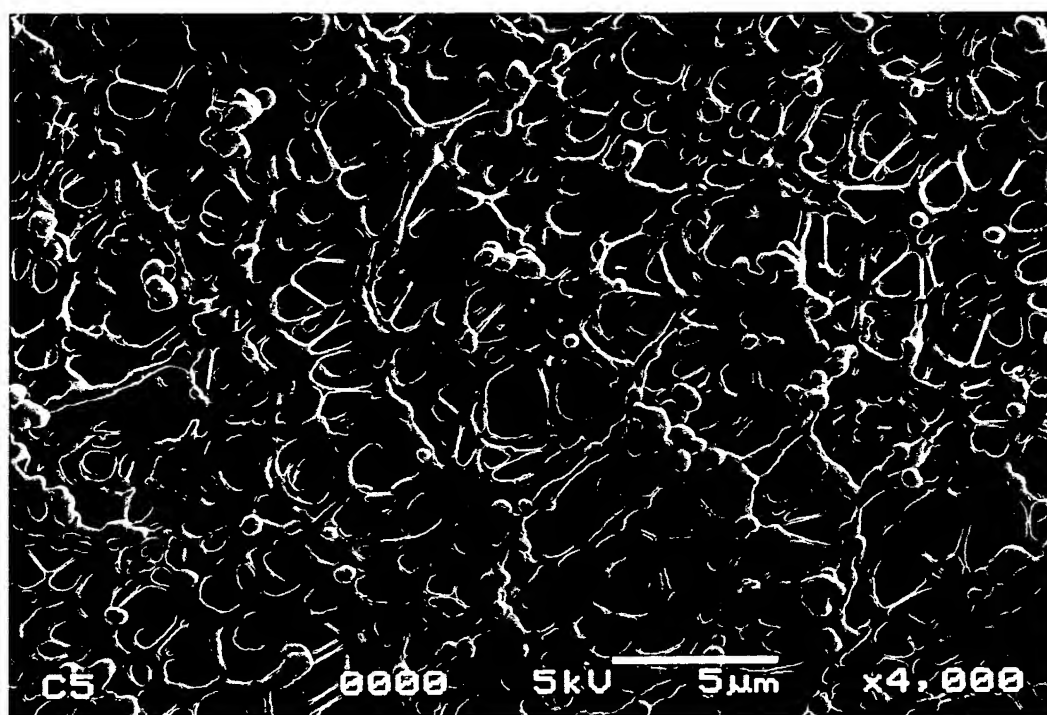
Signed this 24th day of November , 2006



Shyusei Ohya

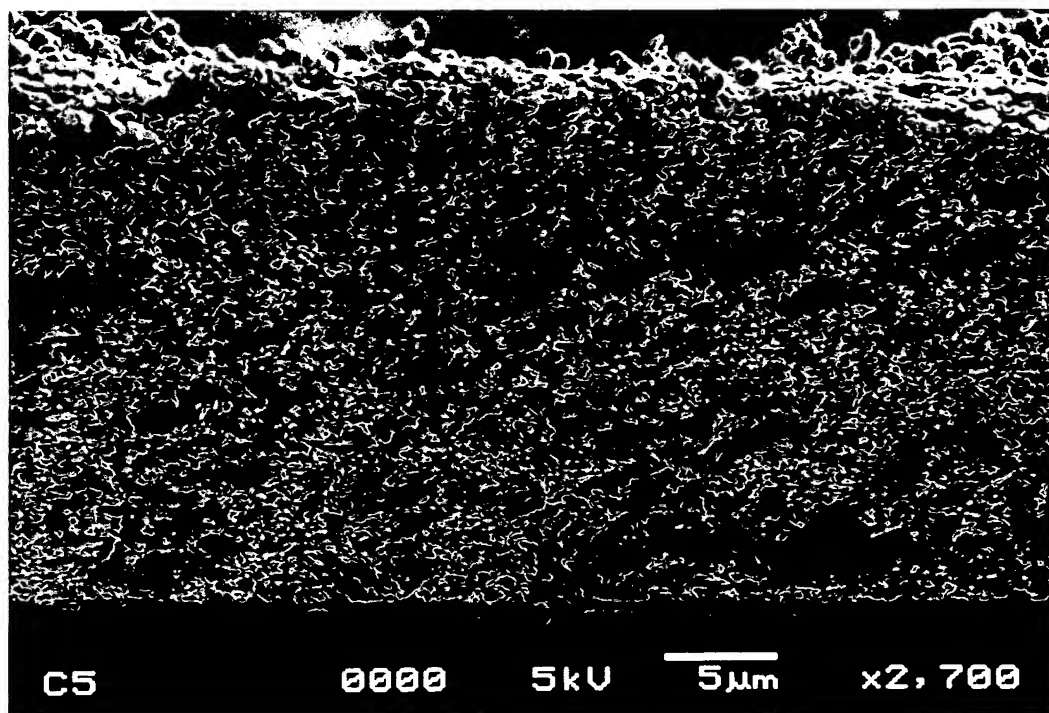


Fig.A



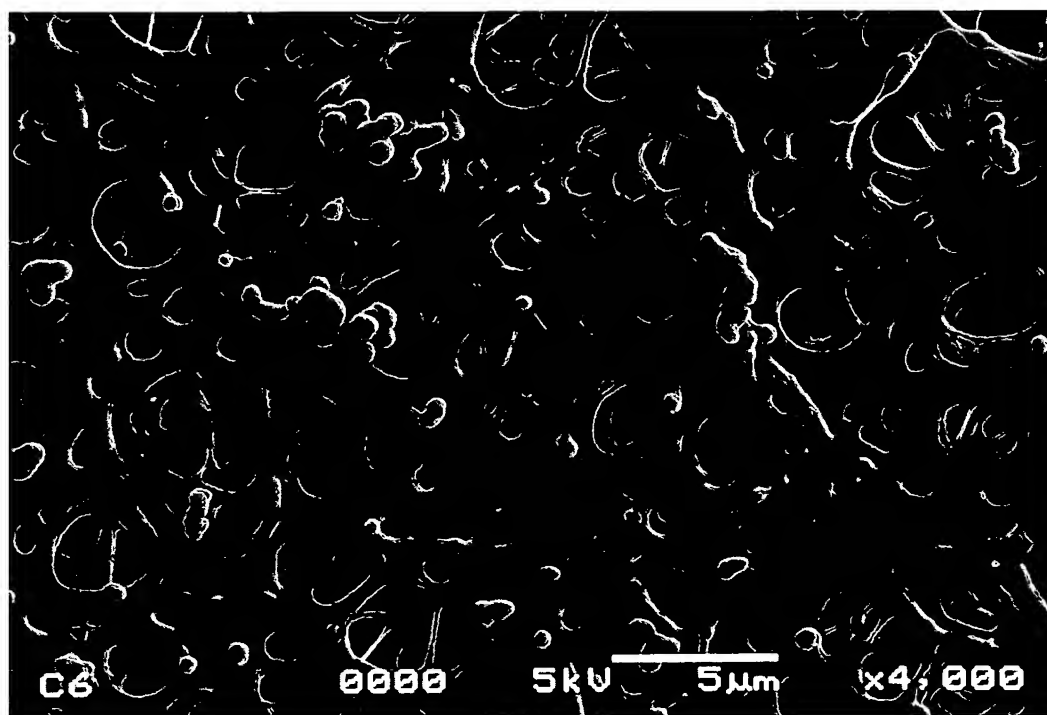
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Fig.B



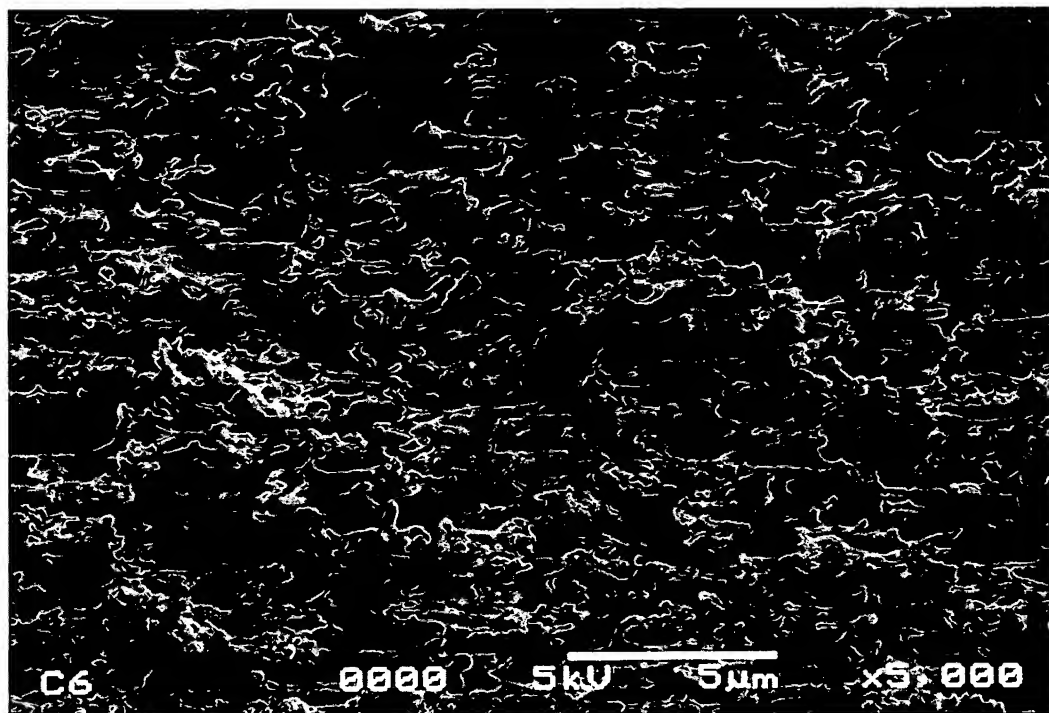
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Fig.C



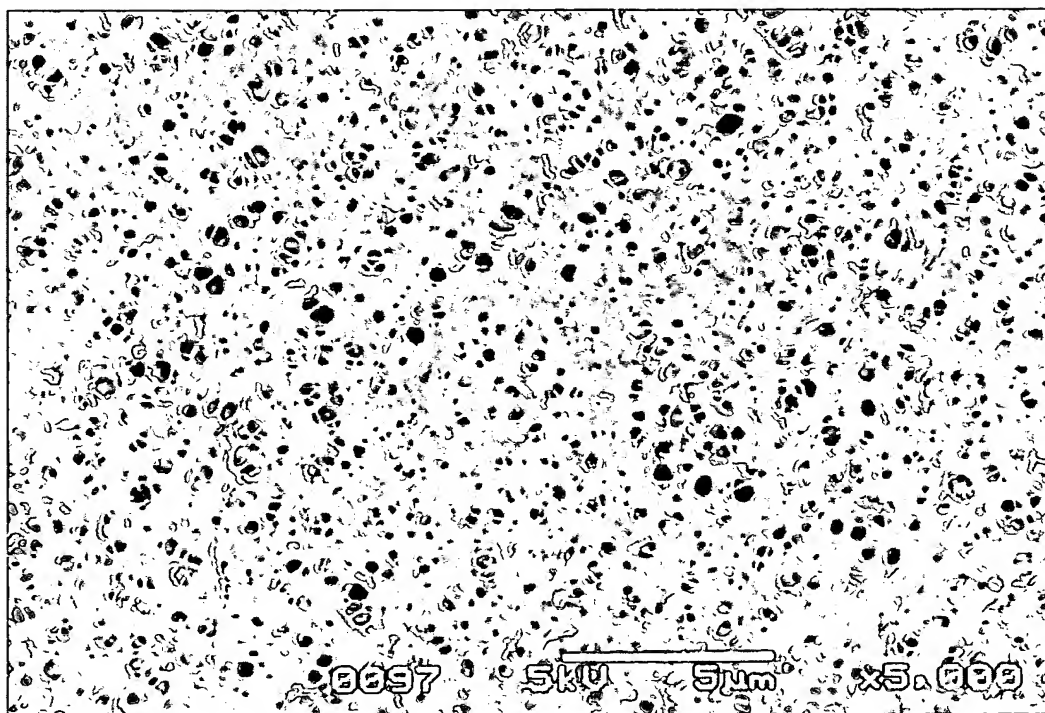
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Fig.D



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Fig.E



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